| ESRF | Experiment title: Investigation of direct elementary reactions of boron with oxygen and hydrogen | Experiment number: HS1082 |
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| Shifts: | Local contact(s): M. Mezouar | Received at ESRF: |

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Report:

This work is a part of Ms. D. Nieto (Phd student, ID30, ESRF)Thesis.

During the first set of experiment, two objectives were fixed:

- Determination of the thermo-elastic behavior and structure evolution of β-boron at high pressure.
- Determination of the boron-oxygen chemistry at high pressure.

# Thermo-elastic properties and structure evolution of β-boron at very high pressure

In spite of its fundamental interest, very few works concerned with this topic are available in the litterature. This lake of informations is mainly due to the experimental difficulties. Indeed, β-boron has a very low X-ray scattering power and a complex crystallographic structure (105 atomes per unit cell). To overcome these difficulties, state-of-the-art techniques only available at 3$^{rd}$ generation sources have been combined to determine the equation of state of β-boron with an unprecedented precision in a wide domain of pressure. High energy (E=33 keV) X-Ray diffraction have been performed on both single crystal and powder of purified β-boron. The quality of the diffraction patterns obtained in this experiment is illustrated in figure 1.

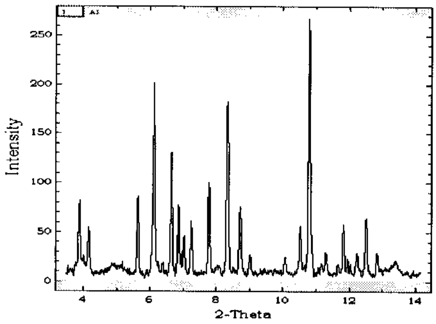


Fig. 1 : Diffraction pattern of β-boron (powder) in a diamond anvil cell

The pressure-volume relation, i.e. the equation of state has been extracted from the data shown in figure 2. Data obtained under non-hydrostatic conditions are also shown in this diagram. They exhibit an important deviations to a "normal" behavior (presence of a large bump). This effect illustrates the fact that very good hydrostaticity conditions, only achieved when helium is used as pressure transmitting medium, are essential. An isothermal bulk moduli of 220 GPa in good agreement with previous ab initio calculations has been obtained.

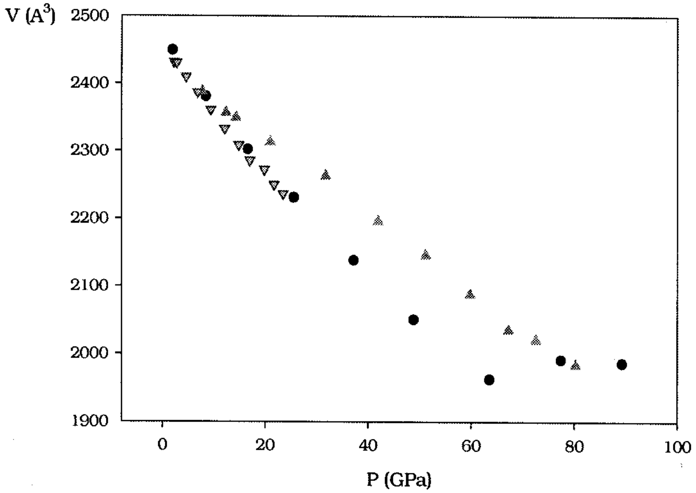


Fig. 2: Pressure-volume data from: Single crystal in helium (circles); Powder in helium (down triangles); Powder in non-hydrostatic conditions (up triangles).

A clear discontinuity, sign of structural phase transition is apparent in the pressure-volume diagram of figure 2, at pressures above 60 GPa. The careful analysis of the diffractograms, obtained from powder data in this pressure region, has confirmed the presence of a phase transformation. However, it was not possible to unambiguously determined the corresponding new structure using only powder data . More experiments using accurate single crystal methods are needed to finalise the structural refinement.

Chemistry of boron-oxygen mixture at high pressure and high temperature

The first set of experiments performed during the run HS1082 has demonstrated the feasibility of pressure-temperature induced chemical reactions in a diamond anvil cell at the ESRF. The chemical reactions in the boron-oxygen system has been systemically studied as a function of pressure. Micro-samples (the sample chamber is a cylinder of 150μm diameter and 50μm thick) of boron and oxygen were untimely mixed and heated at temperatures above 2000 K using the 1.08μm radiation of a YAG laser at different pressures. At all pressures, the reaction was immediate and extremely exothermic (emission of light through the diamonds). After this treatment, the reaction products were analysed in situ using X-ray diffraction at beamline ID30. Diffraction patterns before and after high pressure-high temperature treatment is shown in figure 3. The composition the reaction products was a mixture of residual boron and oxygen, and pure orthorhombic $B_2O_3$; which is a high pressure polymorph of $B_2O_3$. The thermo-elastic behavior (equation of state) of this form of $B_2O_3$ has been determined in situ for the first time.

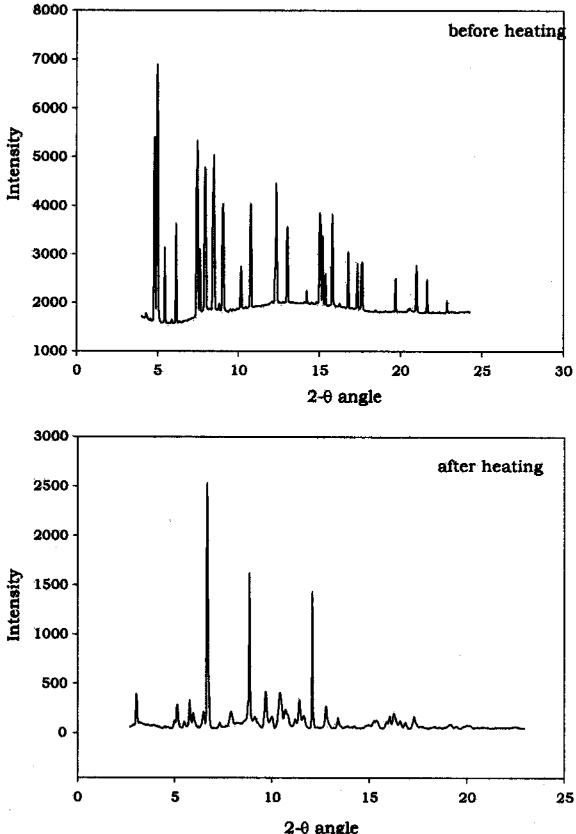
before heating
Intensity
8000
7000
6000
5000
4000
3000
2000
1000
0
5
10
15
20
25
30
2-θ angle
after heating
3000
2500
2000
1500
1000
500
0
2-θ angle

Figure 3